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Effect of calcium lignosulfonate on properties of concrete at early ages

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EFFECT OF CALCIUM LIGNOSULFONATE ON PROPERTIES OF CONCRETE AT EARLY AGES

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A

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ABSTRACT

The objective of this investigation was to determine the effects of calcium lignosulfonate on the physical properties of concrete. Calcium lignosulfonate used as an admixture was tested for its effect on the tensile and compressive strength of plain concrete cylinders, and for its effect on the flexural strength of plain concrete beams. The effect of the admixture on shrinkage, slump, air content and unit weight was also studied.

The test results showed that calcium lignosulfonate reduces the amount of water required to make a workable concrete mix; a more workable mix saves money because it is easier to place, thus reducing labor requirements for the project. Reducing the water content while leaving the cement factor unchanged results in concrete of higher tensile, compressive and flexural strengths at all ages.

ACKNOWLEDGMENT

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The author is also indebted to: Professor James E. Spooner, of the Civil Engineering Department, for his suggestions and experienced advice; Professor R. F. Davidson, Chairman of the Mechanics Department, for making available the facilities of his department's laboratory; Miss Mu-hao Sung, graduating senior student of Taiwan Provincial Cheng Kung University, Republic of China, for outlining the problem for the project.

Sincere thanks are also due to Mr. Kuo-Chu Hu and Mr. Ping-Tung Huang, graduate students at the University of Missouri at Rolla, for their assistance in mixing concrete and taking readings in the laboratory. Without their kind assistance, it would have been impossible to bring this project to a successful conclusion.

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LIST OF SYMBOLS

The symbols used are defined where they first occur in the text and are listed here in alphabetical order for convenience.

A	the cylinder cross-sectional area in square inches
A_1	apparent air content, percentage by volume of concrete
A_2	air content, percentage by volume of concrete
b	average width of specimen in inches
d	diameter of cylinder, in inches
D	average depth of specimen in inches
G	aggregate correction factor, percentage by volume of concrete
h_1	reading at operating pressure
h_2	reading at zero pressure
l	length of cylinder, in inches
L	span length in inches
P	maximum applied load indicated by the testing machine in pounds
R	modulus of rupture in pounds per square inch
S	compressive strength, psi
T	tensile splitting strength, psi

I. INTRODUCTION

1.1 HISTORY

Admixtures for portland cement concrete are those ingredients which are added to the primary constituents (portland cement, aggregates, and water) to improve or modify the properties of the concrete, compensate for some deficiency in a primary constituent, or effect a reduction in cost.*(1) Some materials which are classed as admixtures, such as pozzolan and blood, were concrete ingredients used by the Romans.

Because the addition of another ingredient is very likely to require additional control and technical skill on the part of the concrete producer and inspection agency, and extra facilities for handling and proportioning, and because admixtures in general were originally frowned upon by the cement and concrete industries, these materials were for many years slow in gaining general acceptance. However, the recognition of the role of entrained air in improving frost resistance in concrete has led to almost universal acceptance of the value of air-entraining admixtures and to departure from the previously widely-held concept that all admixtures were of doubtful value.

Some admixtures, such as pozzolans, fly ash, calcium chloride, calcium sulphate and sodium hexametaphosphate have been used for many years, while others are under current development.

A rather limited amount of research has been directed at

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*Numbers in parentheses refer to the bibliography identifying and evaluating the properties of concrete using calcium lignosulfonate as an admixture. It is suspected that calcium lignosulfonate has the effect of increasing the compressive, tensile and flexural strengths of plain concrete at early ages. It is also suspected that calcium lignosulfonate may entrain air. Experiences in the field and laboratories have demonstrated that the addition of a moderate amount of air to any concrete mix will cause a marked increase in resistance to the disintegrating effects of freezing and thawing.(2)

1.2 PROPOSED INVESTIGATION

In this thesis the tests performed were planned to provide sufficient data to establish the value of calcium lignosulfonate as an admixture in plain concrete. This data should answer questions about the effect of the admixture on the strength of concrete as follows:

(1) What is the effect on tensile strength?
(2) What is the effect on compressive strength?
(3) What is the effect on flexural strength?
(4) What is the effect on shrinkage?

In this investigation, four batches of concrete with varying amounts of admixture and water/cement ratios were tested for tensile strength, compressive strength, flexural strength, shrinkage, air content and slump. The tests were performed in the Materials Laboratories of the Civil Engineering Department and the Mechanics Department, University of Missouri at Rolla.

II. REVIEW OF LITERATURE

A number of materials have been produced to be added to concrete to improve certain of its properties. Admixtures may be classified as: Accelerators, retarders, waterproofing agents, finely divided workability aids, pigments, surface active agents and pozzolanas.(3) Many admixtures affect more than one property of the concrete and although they may improve one property they may be harmful to other properties.

Lerch (4) investigated the rates of hydration of neat cement pastes having a water/cement ratio of 0.4 by weight by measuring the rate of evolution of heat by means of a conduction calorimeter. Calcium chloride produced a considerably greater acceleration of the early hydration than plain concrete. The heat of hydration is increased by calcium chloride up to 3 days after mixing. With calcium chloride the greatest acceleration occurred in the first few hours, and the setting time was reduced.

A feature of retarding agents to which much attention has been devoted recently is their ability to reduce the water requirements and to increase the strength of concrete mixes. This subject has been dealt with by Grieb, Werner and Woolf.(5) They experimented with concrete mixes having between 5 1/4 and 6 bags of Type 1 cement per cubic yard, using 1 inch maximum size limestone and natural silica sand aggregates and having an air content of 5 to 6 percent and a slump of 2 to 3 inches. Retarders normally increase the setting time and reduce the subsequent rate of hydration.

The use of pigments in concrete is the subject of British Standard Specification No. 1014 (1942).(6) All pigments must be permanent and in particular it is essential that they should not be affected by the free lime in the concrete. As only a small quantity of some pigments is required, they are sometimes mixed with fillers or extenders. Common materials used for this purpose are chalk and barium sulphate.

A series of tests on the use of air entraining admixtures was conducted by Klieger (7). The main purposes of entraining air in concrete are to increase the workability and to improve the resistance of the concrete to weathering and in particular to the action of frost. The main disadvantage of entrained air is the reduction in the strength of the concrete which it causes.

Calcium lignosulfonate is a suitable dispersing agent which does not interfere with the normal hydration process and does entrain a small amount of air in the concrete.(3) For a fixed ratio of calcium lignosulfonate to cement, the amount of air entrained is fairly constant irrespective of the mix proportions, or at least falls within a much closer range than that which would be obtained with foaming agents. For the amount of dispersing agent which would normally be used, the percentage of air entrained will usually range between 3 and 4 percent; and if higher percentages of air than this are required, a foaming agent should be used. A dispersing agent does not reduce the strength of concrete to the same extent as a foaming agent; it has the same effect as a foaming agent, although possibly to a smaller extent, of increasing the workability and of improving resistance to freezing and thawing and of reducing bleeding.

Tuthill, Adams and Hemme, Jr. have published a paper on the valuation of the compressive strength of concrete which had various lignosulfonate products combined with sugar as an admixture. The paper stated that the compressive strength of concrete.was increased when a nominal dosage of lignosulfonate product was used.(8) It was found that this type of admixture entrains little air. Good performance of such an admixture, at the same cement content and initial slump, included increased compressive strength and lower water requirement. However there was no evidence that the sugar content of lignin-based admixtures should be a cause for concern.

Currently, different types of other admixtures are being improved, both in performance and reliability. In this investigation calcium lignosulfonate was used as an admixture to test for its effect on tensile and compressive strength of plain concrete cylinders, and its effect on the flexural strength of plain concrete beams. The effect of the admixture on the properties of fresh concrete was also studied.

III. TEST APPARATUS

3.1 SIEVES AND SIEVE SHAKER

Two types of electrical sieve shakers were used for screening the fine and coarse aggregate.

(a) The small Rotap shaker, a product of W. S. Tyler Company, which holds six U. S. Standard laboratory sieves at one time, was used to determine the aggregate gradation of the coarse and fine aggregates. Different sieve sizes were used, depending on the type of aggregate.

(b) The larger shaker, a product of Gibson Screen Co., Merser, Pennsylvania, was used for grading the normal and lightweight coarse aggregates prior to mixing of the concrete. Its wire mesh screens are of the following grid opening sizes: 1", 3/4", 1/2", 3/8", No. 4 and No. 8.

3.2 CONCRETE MIXER

The Lancaster Counter Current Batch Mixer, three cubic foot capacity, was used in the preparation of the experimental concrete. This mixer, shown in Figure 1, is manufactured by the Lancaster Iron Works of Lancaster, Pennsylvania.

3.3 CYLINDER MOLDS

Paraffin covered, standard 6" paper cylinder molds were used to cast the concrete cylinders. These metal-bottom molds are produced by the Tillery Container Corporation of Kansas City, Missouri.

Figure 1. Concrete Mixer.

3.4 AIR ENTRAINMENT PRESSURE METER

The air-entrainment pressure meter, shown in Figure 2, consisted of a portable device made from an alkali-resistant lightweight alloy. It is known as the ACME Air-entrainment Meter which is manufactured by E. W. Zimmerman, Chicago, Illinois. It has three main parts: the calibration cylinder, the bowl, and the upper assembly.

3.5 COMPRESSION TESTING MACHINE

Tensile and flexural specimens were tested on a Riehle Hydraulic Compression Testing Machine of 0-60,000 and 0-300,000 pounds capacity. The lower range was used for the flexural specimens and the upper range for tension cylinders. This machine, shown in Figure 3, is located in the laboratory of the Mechanics Department of the University of Missouri at Rolla. Calibration of the test machine was done by a Riehle representative and it was checked by members of the Mechanics Department staff and found to be within the allowable tolerance of one percent.

The Forney Hydraulic Compression Testing Machine shown in Figure 4 was used for testing the compressive strength of cylinders. It has two ranges: Low range, 0-60,000 pounds and high range, 0-300,000 pounds. It is manufactured by Forney, Inc., Pennsylvania.

3.6 MICROMETER SCREW GAUGE

The micrometer screw gauge shown in Figure 5 is twenty-five inches long, and is capable of being read to the nearest one thousandth of an inch. It is a product of Brown and Sharpe Manufacturing Corporation, Providence, Rhode Island.

Figure 2. Air Entrainment Pressure Meter.

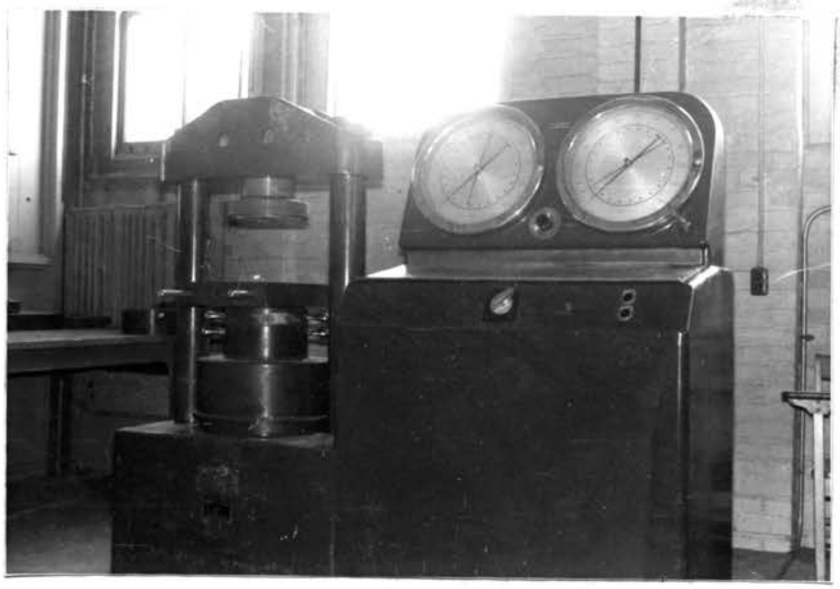


Figure 3. Riehle Compression Testing Machine.

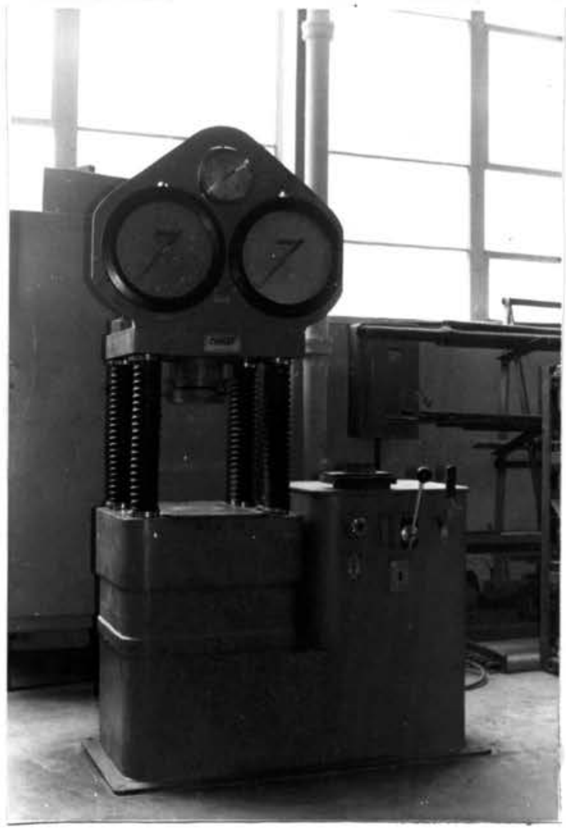


Figure 4. Forney Compression Testing Machine.

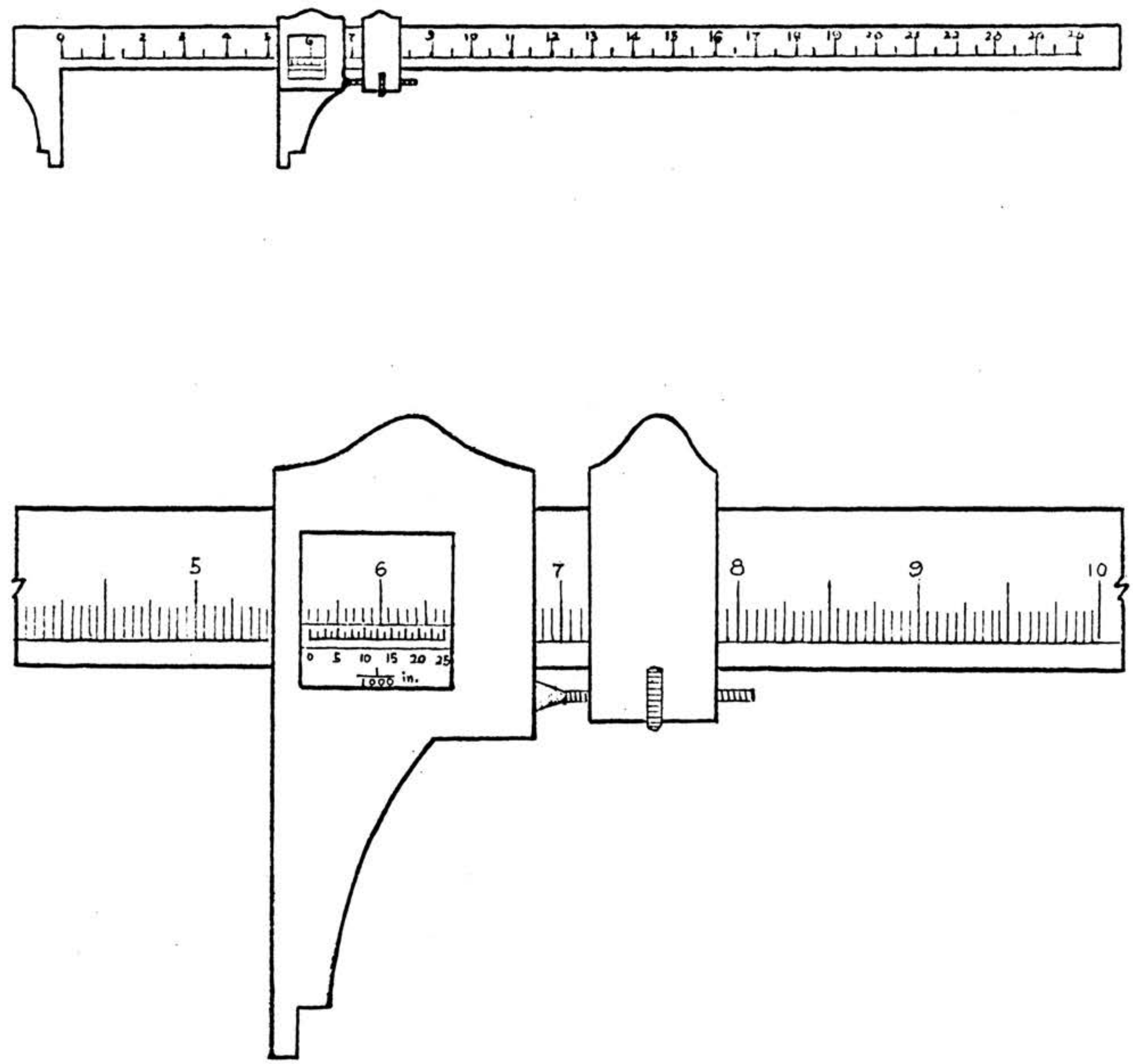


Figure 5. Micrometer Screw Gauge.

IV. MATERIALS USED IN TESTS

4.1 INTRODUCTION

For preparing the specimens, the following materials were used: (a) cement, (b) water, (c) fine aggregate, (d) coarse aggregate, and (e) the admixture, calcium lignosulfonate.

4.2 CEMENT

In all mixes the cement used was commercial portland cement Type 1. The cement was manufactured by Marquette Portland Cement Company, Cape Girardeau, Missouri. Its specific gravity was determined to be 3.15.

4.3 WATER

Mixing water was procured from the system of the University of Missouri at Rolla.

4.4 FINE AGGREGATE

The fine aggregate used was river sand procured from the Meramec River Sand and Gravel Company, Pacific, Missouri. The specific gravity and absorption were determined in the laboratory in accordance with the "Method of Test for Specific Gravity and Absorption of Fine Aggregate", ASTM Designation C128-59, and were found to be: (a) specific gravity: 2.55, (b) absorption: 1.42%.

The standard method of test for "Sieve Analysis of Fine Aggregate," ASTM Designation C136-46 was followed. Table I shows the tabulated results of the sieve analysis and Figure 6 shows the limits of the specifications and a plot of the sand gradation curve. It can be noted from Figure 6 that the gradation of the fine aggregate is within the prescribed limits. This fine aggregate has a Fineness Modulus of 2.60.

4.5 COARSE AGGREGATE

All mixes contained coarse aggregate from Springfield, Missouri, supplied by C. F. Farney of Rolla. The coarse aggregate was 3/4" maximum size, crushed white limestone. The average specific gravity of the coarse aggregate was found to be 2.65, and its absorption was found to be 0.497%, in accordance with the "Method of Test for Specific Gravity and Absorption of Coarse Aggregate," ASTM Designation C127-59.

The standard method of test for "Sieve Analysis of Coarse Aggregate," ASTM Designation C136-61T, was followed. Table 2 shows the tabulated results of the sieve analysis and Figure 7 shows the limits of the specifications and a plot of the coarse aggregate gradation curve. The results indicated that "as delivered" coarse aggregate was satisfactory to meet ASTM 3/4" aggregate gradation requirements, therefore no sizing procedure was required.

4.6 ADMIXTURE-CALCIUM LIGNOSULFONATE

The admixture used was calcium lignosulfonate with the brand name "W-88-C". It was manufactured and delivered in powder form by the Burtonite Company, Nutley, New Jersey. It is a yellow powdered substance which is water soluble.

Table I. Sieve Analysis for Fine Aggregate

Sieve Size	Weight Retained	Percent Retained	% Retained (Accum.)	Percent Passing
3/8"	0.0	0.0	0.0	100.0
No. 4	1.0	0.1	0.1	99.9
No. 8	95.1	9.5	9.6	90.4
No. 16	193.2	19.3	28.9	71.1
No. 30	152.0	15.2	44.1	55.9
No. 50	338.8	33.9	78.0	22.0
No. 100	212.9	21.3	99.3	0.7
Pan	7.0	0.7	----	0.0
Total	1000.0	100.0	----260	----

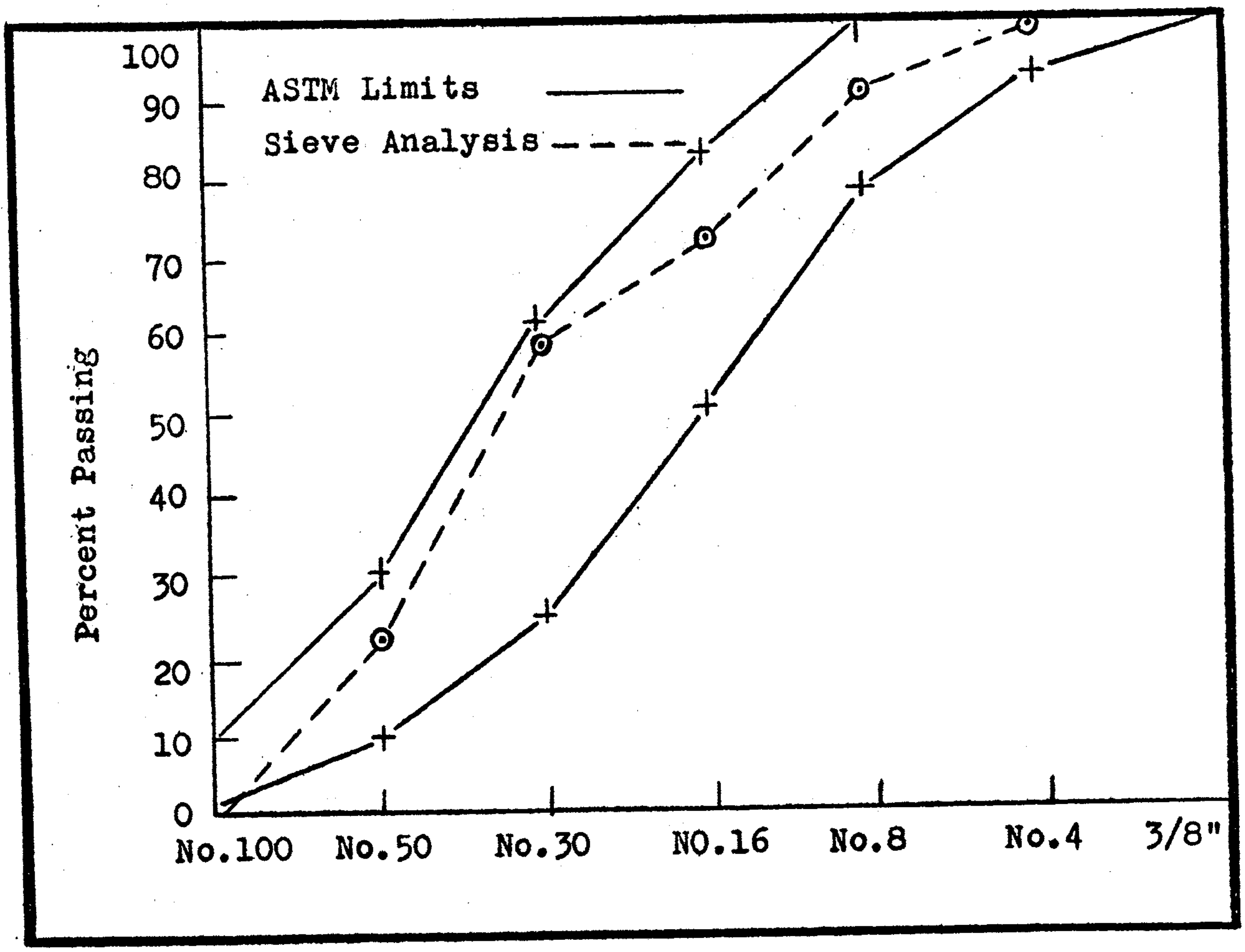


Figure 6. Gradation Curve for Fine Aggregate

Table II. Sieve Analysis for Coarse Aggregate

Sieve Size	Weight Retained	Percent Retained	% Retained (Accum.)	Percent Passing
1"	0.0	0.0	0.0	100.0
3/4"	2.5	4.9	4.9	95.1
3/8"	34.0	65.6	70.5	29.5
No. 4	13.8	26.8	97.3	2.7
No. 8	1.4	2.7	100.0	0.0
Pan	0.0	0.0	-----	0.0
Total	51.7	100.0	-----	-----

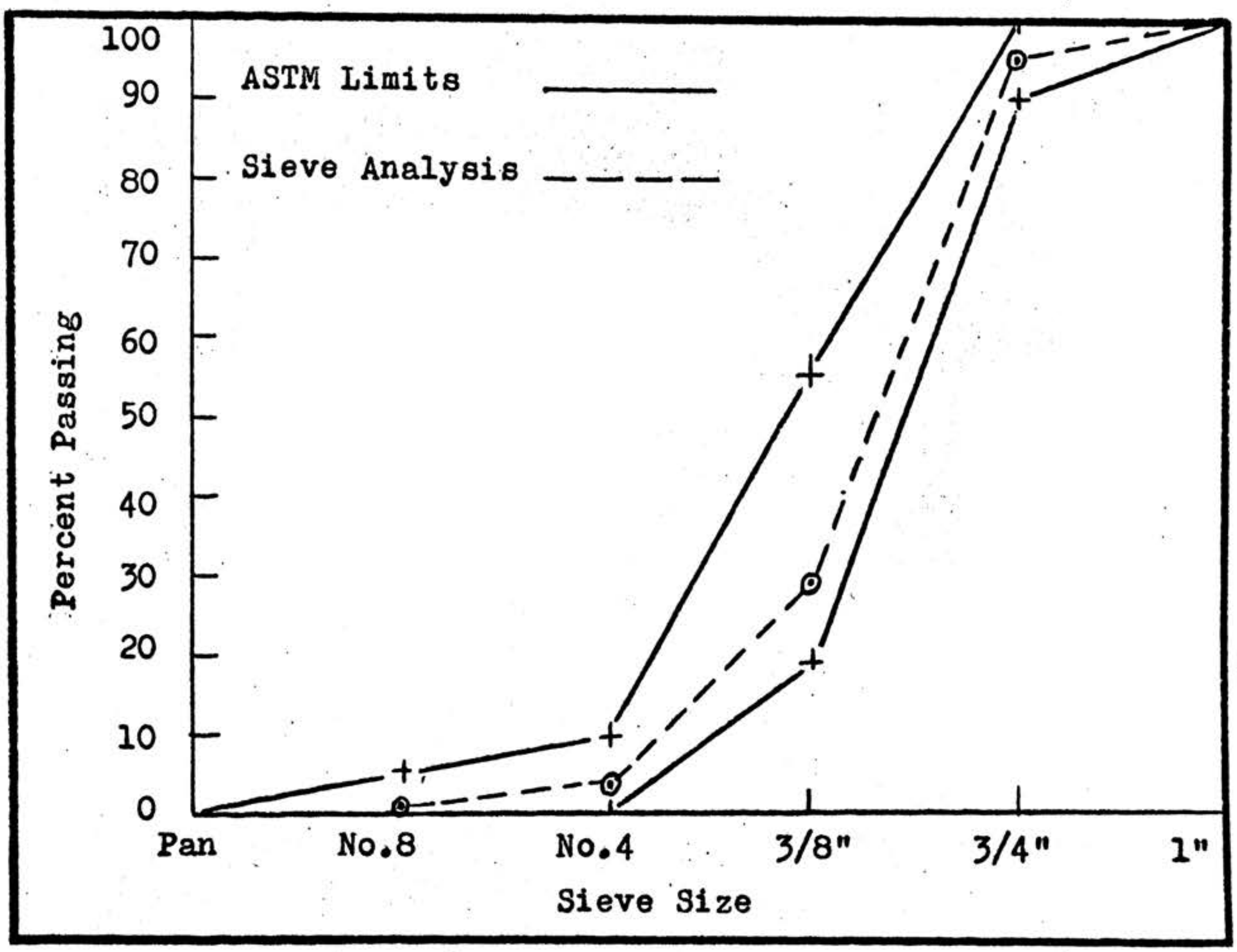


Figure 7. Gradation Curve for Coarse Aggregate

Lignosulfonates by chemical definition are water-soluble, surface-active derivatives of lignin. They are hetero-disperse polymers whose molecular weights vary between 1,000 and 2,000. The organic structure of lignosulfonate compounds has not been completely determined, but it is known that the basic lignin monomer unit is a substituted phenyl propane. A section of the polymeric lignosulfonic acid could have the structure shown in Figure 8.(9)

The admixture used has the following physical characteristics:

Moisture Content	7%
Ash Content	8%
Caloric Value	7,830 BTU/lb.
pH of 1 % Water Solution	4.5
Particle Size	All through 60 mesh sieve
Specific Gravity of 50% Water Solution	1.27 -- 31 Degrees Beaume
Bulk Density	31 lbs./cu. ft.

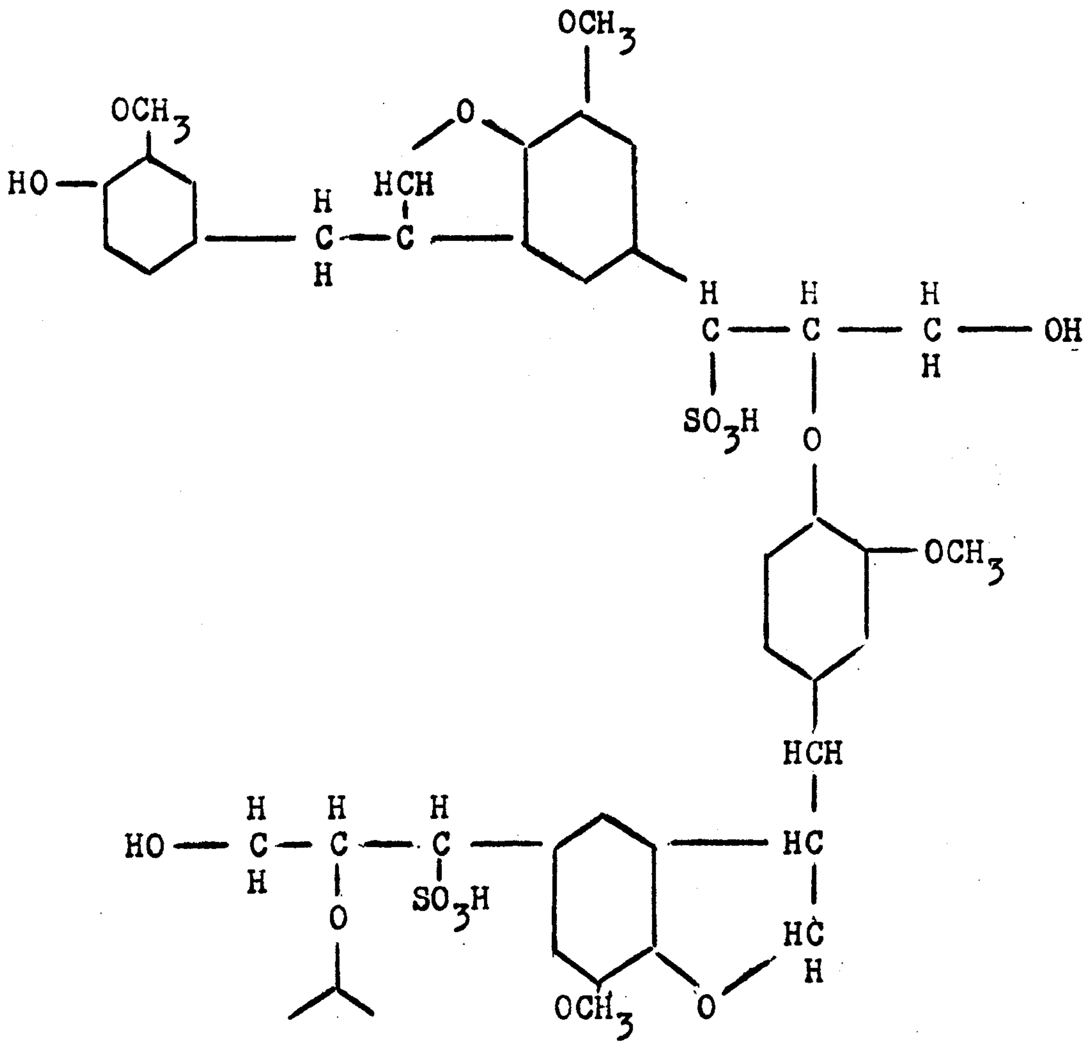


Figure 8.
The Structure of a Section of the Polymeric Lignosulfonic Acid

V. TEST PROCEDURE

5.1 INTRODUCTION

There were six test procedures involved in this investigation.

(a) Preparation of specimens.
(b) Tests for properties of fresh concrete.
(c) Test for tensile strength.
(d) Test for compressive strength.
(e) Test for flexural strength.
(f) Test for shrinkage of concrete.

Each test will be discussed in detail in subsequent paragraphs.

5.2 PREPARATION OF SPECIMENS

(a) Design of Mixes: There are several satisfactory methods of designing a concrete mix; however, the one selected was the procedure outlined by the Portland Cement Association. (10) This procedure is based upon the relationship of the water/cement ratio to compressive strength and is based on the absolute volume criteria. The combination of coarse aggregate, fine aggregate, cement and water was selected which gave the desired compressive strength and 3 inches slump. From the data shown in the P. C. A. Design Manual, and with the necessary calculations, the final mix design was obtained. The complete design with computations is shown in Appendix A and Appendix B. The net yield for each mix was 2.8 cubic feet per batch. The admixture, calcium lignosulfonate, was dissolved in water and replaced an equal amount of water. The rate at which admixture was added to water was slow enough for the particles to separate in agitated water without lump formation.

(b) Batching and Mixing: The constituents for the experimental concrete mixes were proportioned by weight and then mixed for one minute.

(c) Casting and Curing: In accordance with "Standard Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Laboratory", ASTM Designation C192, the cylinders were filled by thirds and rodded 25 times at each layer. The excess concrete was struck from the top of the cylinders and the specimens were placed in the curing room. The curing operation was accomplished by means of a curing room which maintained a relatively constant air temperature, 70 degree F. and a continuous water fog, 100 percent relative humidity. At the end of one day the paper cylinder molds were removed from the specimens to permit uniform curing.

5.3 TESTS FOR PROPERTIES OF FRESH CONCRETE

Slump tests were made on each batch immediately after mixing and before placing the concrete in the cylinder molds or beam forms. Slump tests were conducted in accordance with "Standard Method of Test for Slump of Portland Cement Concrete", ASTM Designation C143-58. The slump was recorded in terms of inches to the nearest 1/4 inch of subsidence of the specimen during the test as follows:

Slump = 12 - Inches of Height After Subsidence

The unit weight of the fresh concrete was determined by measuring the weight of the cylinder molds before and after placing the concrete in the cylinder molds.

Air content tests were conducted in accordance with "Standard Method of Test for Air Content of Freshly Mixed Concrete by the Pressure Method," ASTM Designation C231-60. The air content of the concrete was calculated as follows:

$$A_2 = A_1 - G$$

where: A_1 - Apparent Air Content, Percentage by Volume of Concrete.

A_2 - Air Content, Percentage by Volume of Concrete.

G - Aggregate Correction Factor, Percentage by Volume of Concrete.

The illustration of pressure method of test for air content is shown in Figure 9.

5.4 TEST PROCEDURE FOR TENSILE STRENGTH

The various shapes of specimens used in direct tension tests have all suffered from stress concentrations, and their making and testing have been comparatively difficult processes requiring considerable skill and care. Effort was made to develop and utilize a new method for the determination of the tensile strength. Such a new test was introduced by Mr. F. Carniero, of Brazil (11) and it was decided to include this test in the current series. Mr. Carniero's method utilized a compressive load applied to a cylinder along two opposite platens. The loading arrangement for an indirect tensile test is shown in Figures 10 and 11. Figure 12 shows that the failure of cylinder occurs along the center line.

Tests were conducted on a Riehle Hydraulic Compression Testing Machine with a capacity of 0-60,000 and 0-300,000 pounds.

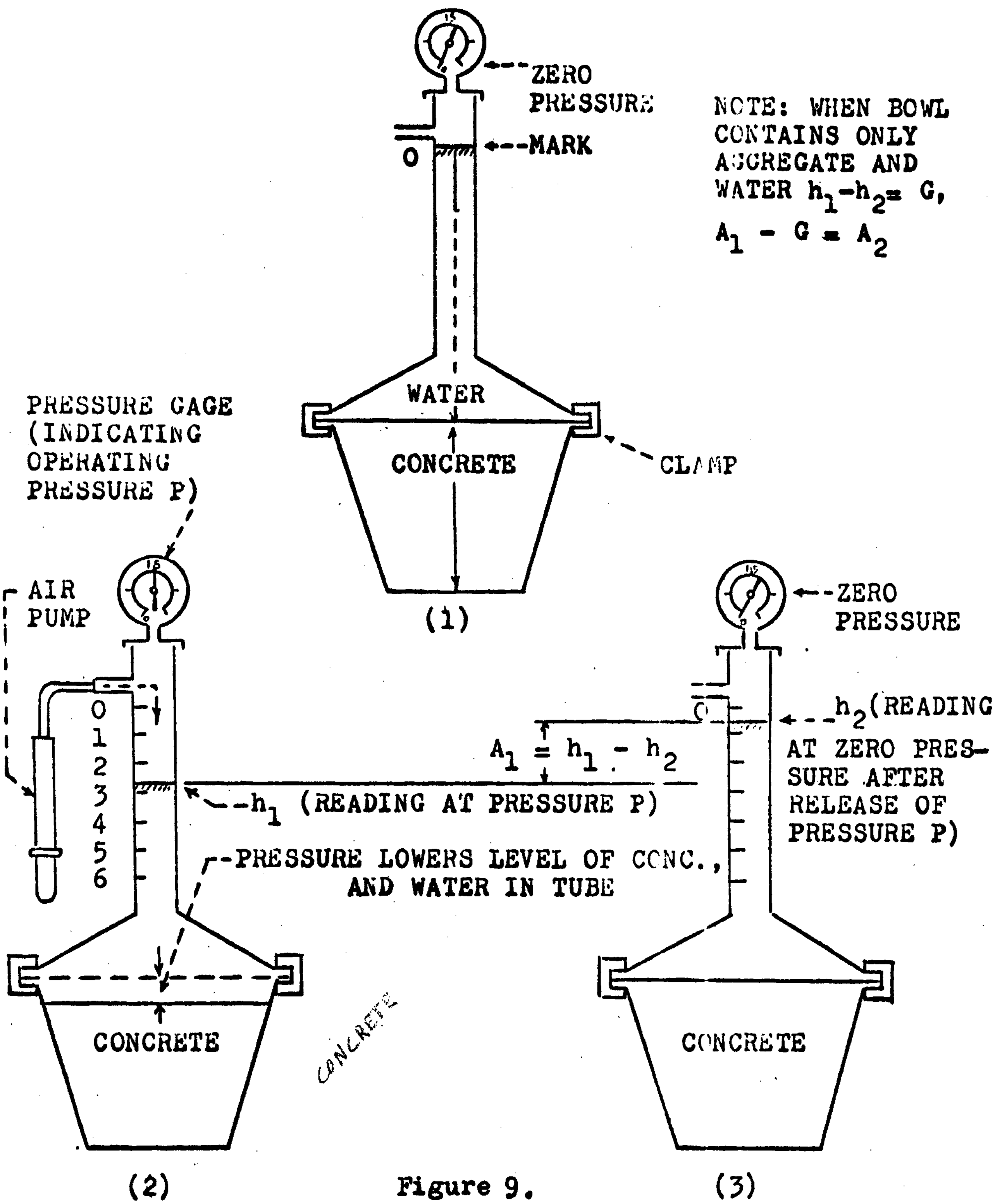


Figure 9.

Illustration of Pressure Method of Test for Air Content

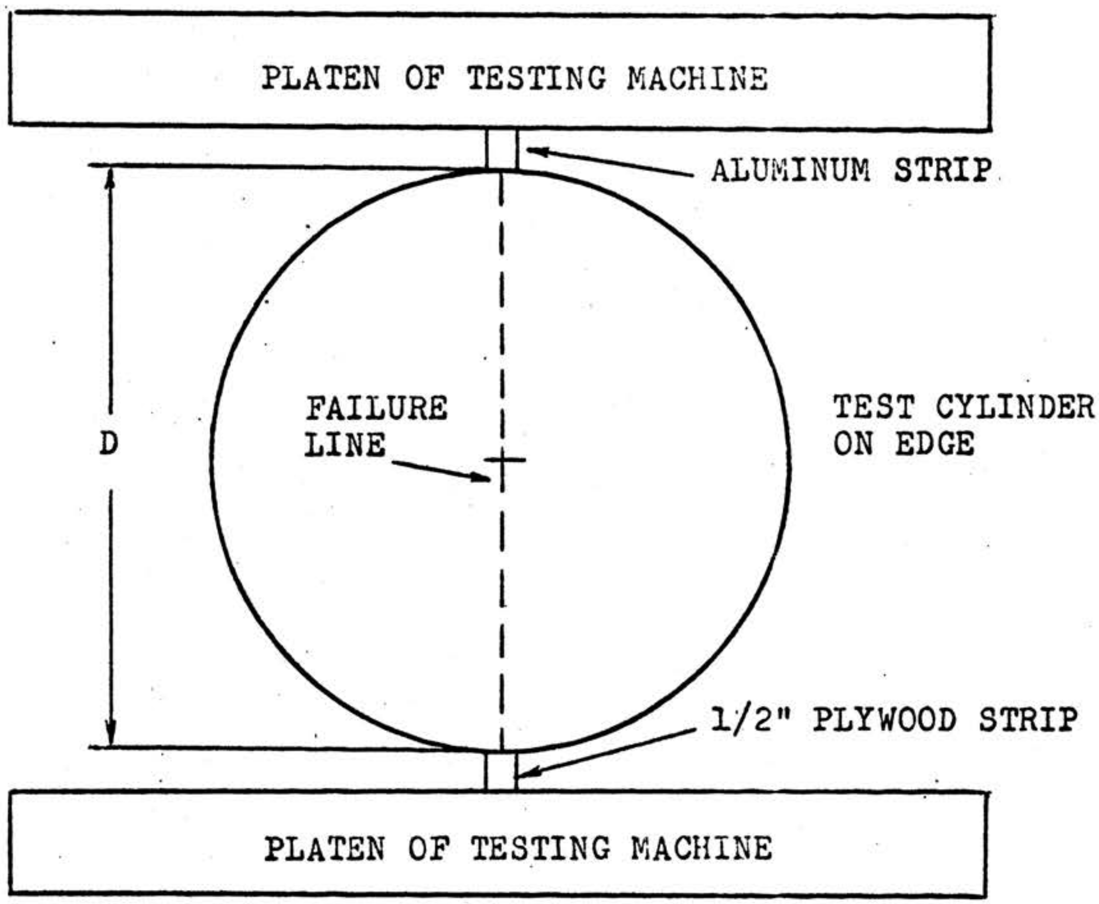


Figure 10. Loading Arrangement for Indirect Tensile Test

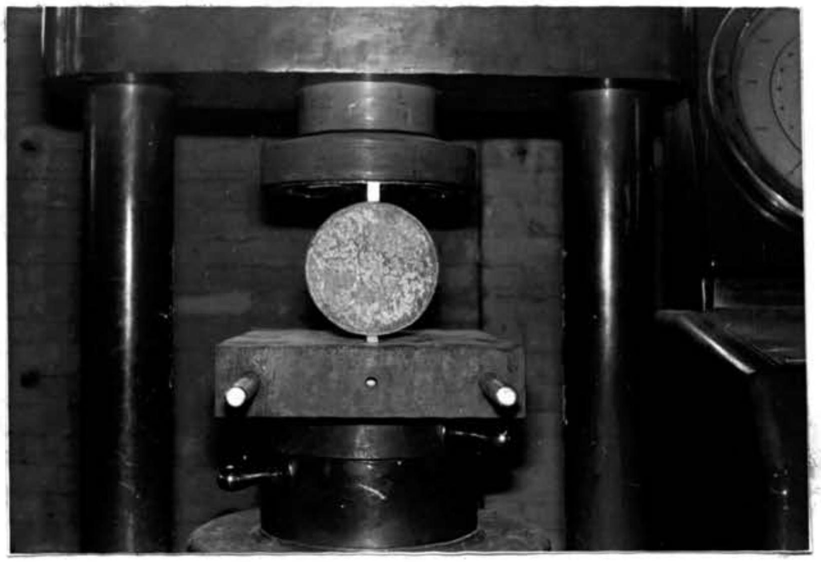


Figure 11. Cylinder Under Indirect Tensile Test.

Figure 12. Cylinder Failure Along the Center Line.

Strips of aluminum and plywood were placed between the platens of the machine and the specimen to obtain the desired results. Tests were scheduled on specimens ranging in age from three days to twenty-eight days. The forming and pouring of cylinders for this series was done as described in section 5.2.

Testing procedure was as follows:

(a) Remove form from cylinder
(b) Place cylinder on testing machine platen
(c) Align the aluminum and plywood strips
(d) Check strip alignment to insure diametral opposition
(e) Apply load to specimen
(f) Record ultimate load.

The loading rate used was determined from the speed at which readings of the gages and machine could be made and recorded. When this rate was determined, it was used throughout the tests. The loading rate used was 3000 psi per minute.

The theoretical analysis of this loading condition shows the uniform tensile stress developed to be:

$$T = 2P / dl\pi$$

where: T - Tensile Splitting Strength in psi
P - Load at Rupture in pounds
d - Diameter of Cylinder in inches
l - Length of Cylinder in inches

5.5 TEST PROCEDURE FOR COMPRESSIVE STRENGTH

The compression tests were performed on standard 6" by 12" cylinders cast in paraffined paper molds with metal bottoms. The forming and pouring of cylinders was done in the same manner as for the tensile series. Tests were planned at ages ranging from three days to twenty-eight days. Specimens were tested on a Forney Hydraulic Compression Testing Machine. The loading arrangement for compressive test is shown in Figure 13.

The testing procedure was as follows:

(a) Remove form from specimen
(b) Cap cylinder after 48 hours
(c) Place cylinder in testing machine
(d) Apply load
(e) Record ultimate load

Since the cylinders are short columns, they were considered to develop a uniformly distributed stress through a transverse section of the cylinder. From this assumption the unit stress, S, is given by the equation

$$S = P / A$$

where: P - The Axial Load in pounds

A - The Cylinder Cross-sectional Area in square inches

5.6 TEST PROCEDURE FOR FLEXURAL STRENGTH

Specimens for the flexural strength tests were 3 1/2" by 4 1/2" by 18" beams. The flexural tests were made in accordance with the "Method of Test for Flexural Strength of Concrete (Using Simple Beam With Center-Point Loading)," ASTM Designation C293-59. The specimens were tested at ages of three, seven, fourteen and twenty-eight days on the Riehle Hydraulic Compression Testing Machine. The load range 0-60,000 pounds was used for all the flexural tests. All beams were tested to failure and the ultimate load was noted. The failure took place at the center line of the beam in all the tests.

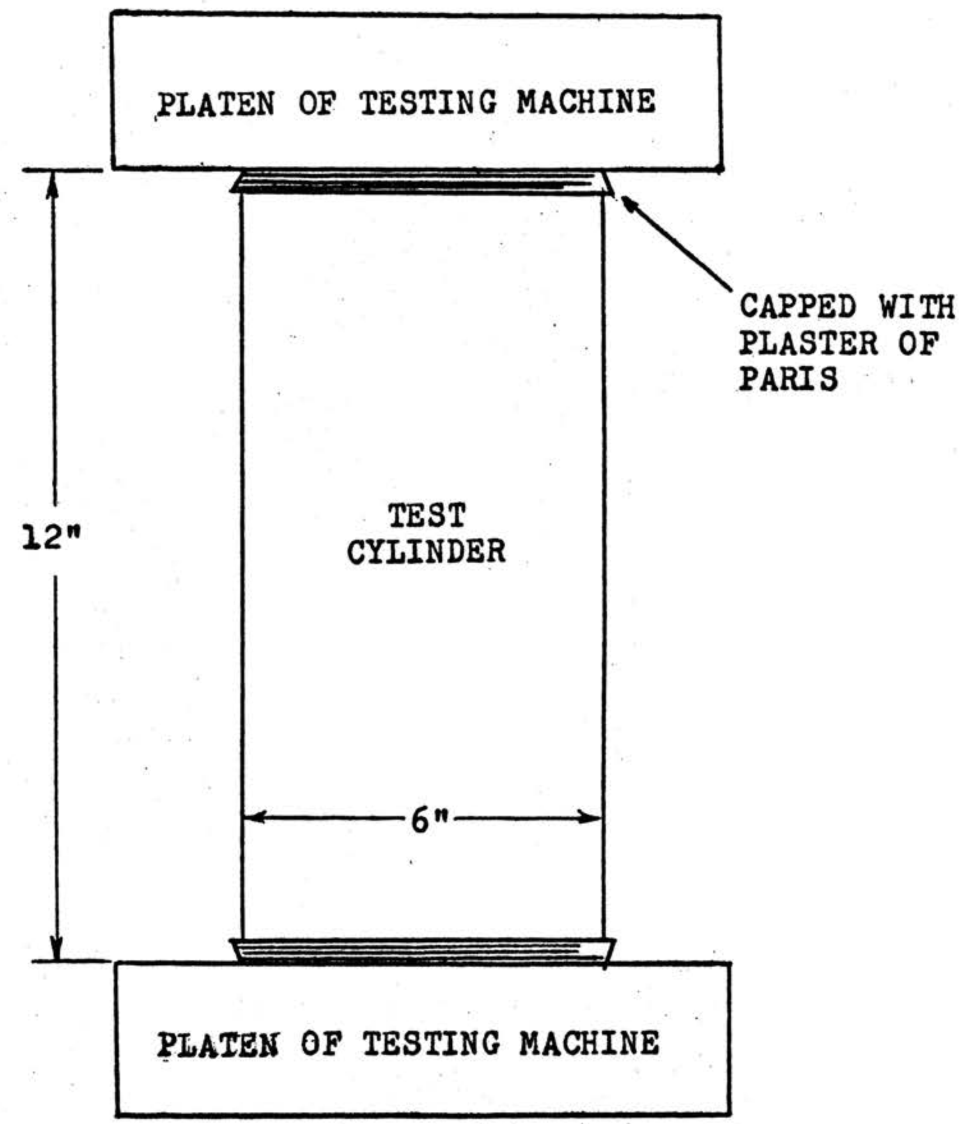


Figure 13. Loading Arrangement for Compressive Test

The testing procedure was as follows:

(a) Remove form from specimen

(b) Place specimen on supports

(c) Place testing head in position

(d) Zero test machine

(e) Load to failure

(f) Record ultimate load and observe position of failure point.

The weight of the beam was neglected in calculations of the modulus of rupture. The modulus of rupture is defined as the maximum flexural stress developed in a beam at the time of failure. The modulus of rupture was calculated as follows:

$$R = 3PL / 2bD^2$$

where: R - Modulus of Rupture in pounds per square inches

P - Maximum Applied Load Indicated by the Testing Machine in pounds

L - Span Length in inches

b - Average Width of Specimen in inches

D - Average Depth of Specimen in inches.

A diagrammatic view of the apparatus for the flexure test of concrete by the Center-Point Loading Method is shown in Figure 14. Figure 15 shows a beam which is loaded under a Center Point Flexure Test. Figure 16 shows that the failure of the beam occurs in the center of span.

5.7 TEST FOR SHRINKAGE OF CONCRETE

The tests for shrinkage were made on 4" by 4" by 11" beams in accordance with "Method of Test for Volume Change of Cement Mortar and Concrete," ASTM Designation C157-60T. Nine beams were cast from three batches of concrete each

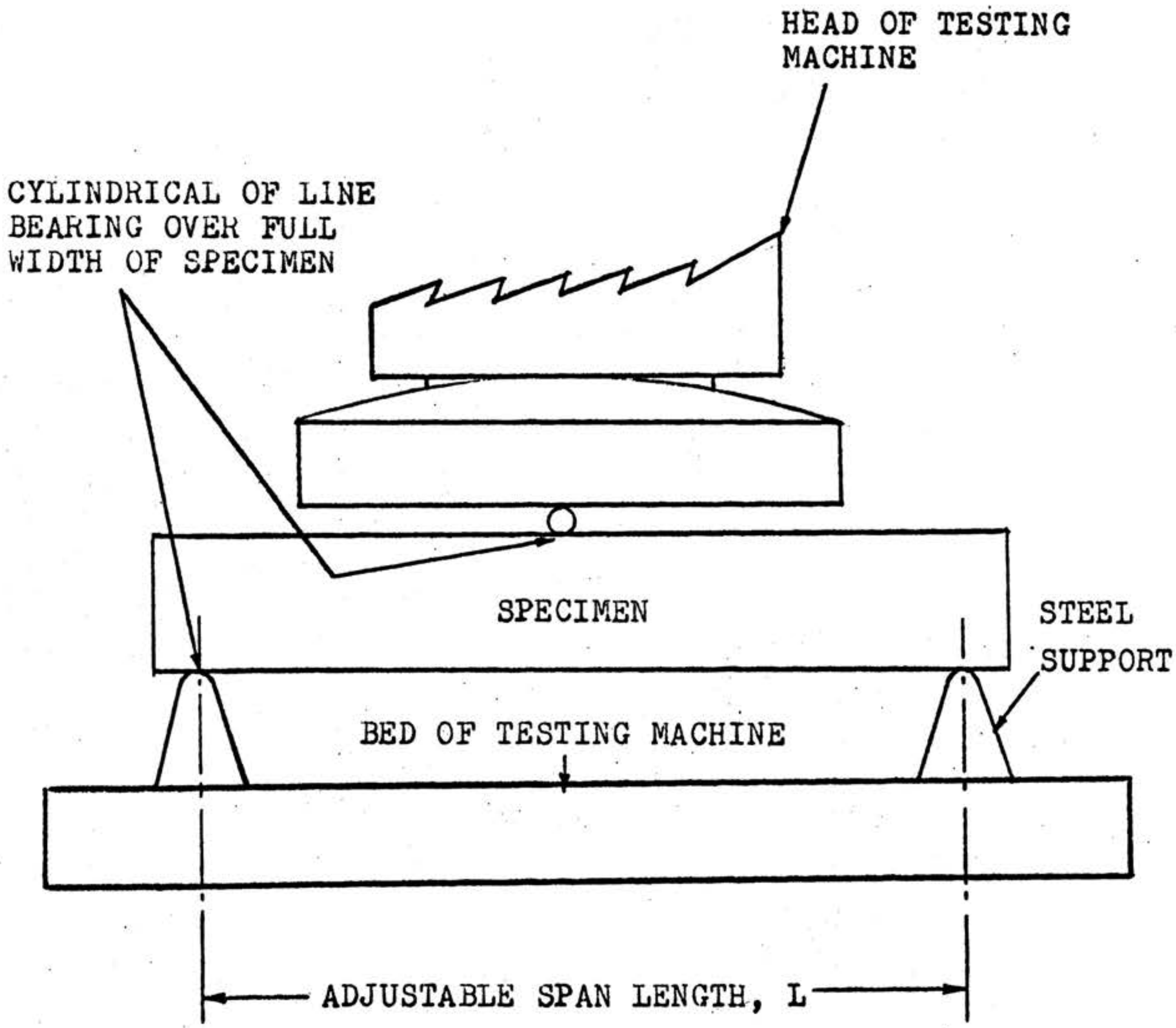


Figure 14. Diagrammatic View of Apparatus for Flexure Test of Concrete by Center-Point·Loading Method.

Figure 15. Beam Under Center Point Flexure Test.

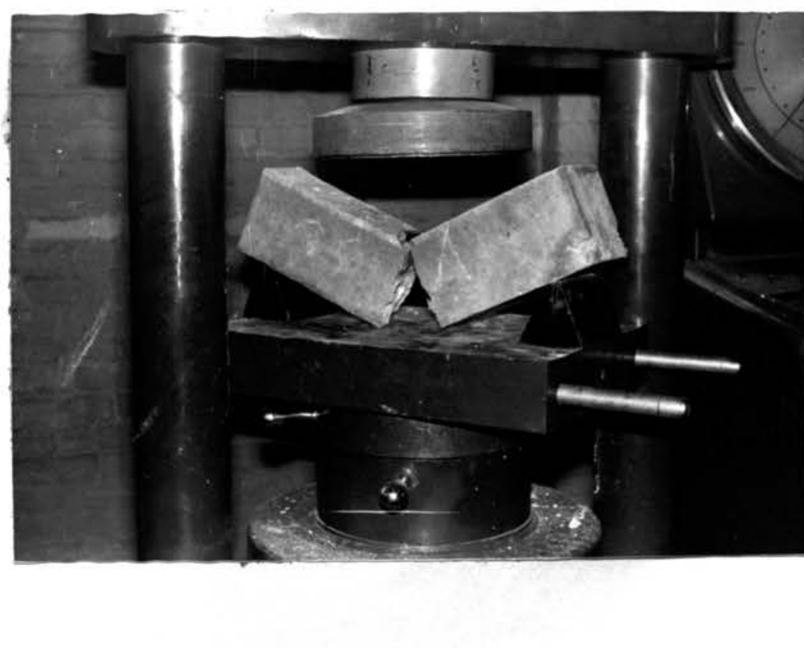
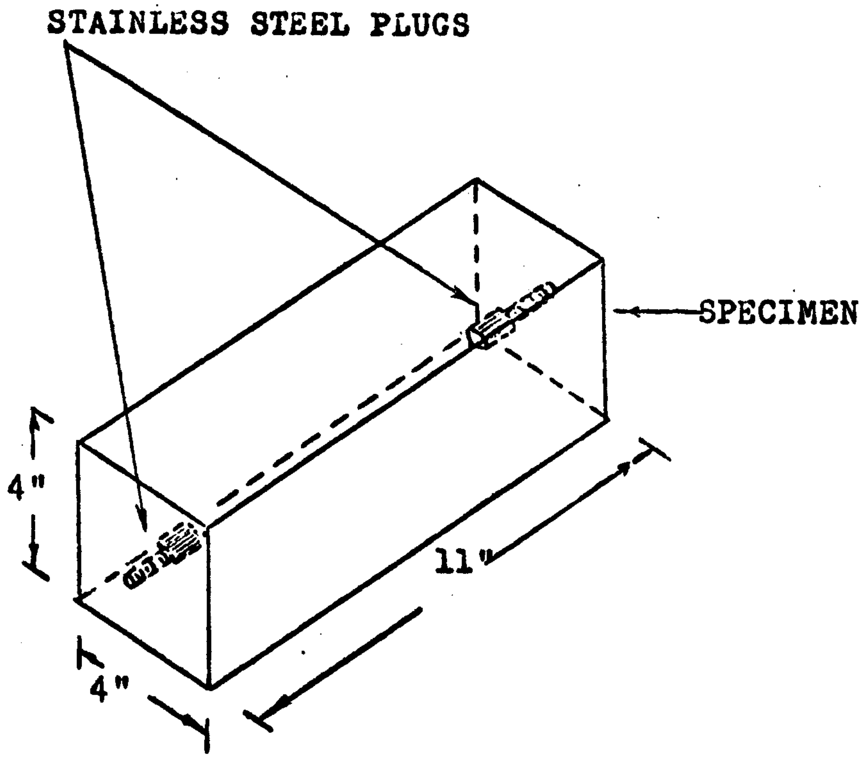


Figure 16. Beam Failure in the Center of Span.

with a different percentage of calcium lignosulfonate as the admixture. Specimens were cast in steel molds and immediately placed in the moist room which was maintained at 70 degree F. and 100 percent relative humidity. After twenty-four hours, the molds were removed and the lengths measured. The specimens were then cured for twenty-eight days in the moist room and a second measurement of length was made. The specimens were then stored outside the moist room in laboratory air for seven days and the lengths measured.

The lengths of the specimens were measured with a micrometer screw gauge capable of measuring to the nearest one thousandth of an inch.

The shrinkage specimens had stainless steel plugs cast at the center in each end as shown in Figure 17.



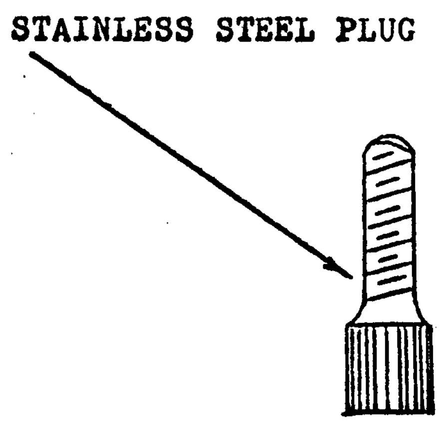


Figure 17. Shrinkage Test Specimen

VI. TEST RESULTS AND DISCUSSION

6.1 PROPERTIES OF FRESH CONCRETE

The properties of fresh concrete tested were slump, unit weight, air content and water/cement ratio. The results are shown in Table 3. In this investigation, Mixes A, C and D were made with same water/cement ratio, 0.452; Mixes A and B were made with same slump, 2.9".

Table 3 shows that the slump of the fresh concrete increased with increasing amounts of admixture if the water/cement ratio was kept constant, 0.452. The slump of Mixes A, C and D was 2.9", 5.2" and 7.0" respectively. Normally the slump is directly proportional to the water/cement ratio of a mix. The reason the slump increased with the increased amount of admixture may be due to the increased air content. For Mix B, the water/cement ratio was reduced to keep the slump the same when the admixture was added and the resulting concrete was still more workable than the concrete of the same slump but with no admixture.

There was an increase in air content with an increase in percentage of admixture. The average air content for Mixes A, B, C and D was 1.1, 1.2, 1.7 and 2.6 percent respectively. This admixture can entrain a little air over plain concrete.

The unit weight of the concrete remained fairly constant from Mix A to B while the unit weight decreased from Mix A to C and D. Part of the change is probably due to the increased air content.

Table III.

Properties of Fresh Concrete

Mix	Percentage Admixture Used	Water Cement Ratio	Unit Weight in lb/cu.ft.	Slump in inches	Percentage Air Content
A	0.00	0.452	145.2	2.9	1.1
B	0.25	0.407	145.0	2.9	1.2
C	0.25	0.452	144.2	5.2	1.7
D	0.50	0.452	142.8	7.0	2.6

6.2 TENSILE STRENGTH TEST

All laboratory data and the computed results of tensile strength test are shown in Table IV. Figure 18 shows the relation between tensile strength and age for each mix, and Figure 19 shows the relation between tensile strength and mix for each age.

From Figure 18, it can be seen that strength-age curves of all mixes look like parabolas. In comparing the 28 days strength of the three mixes A, C and D, it is seen that as the amount of admixture increases, tensile strength increases except at 28 days, when Mixes C and D fall below Mix A. Mixes A, C and D were made with same water/cement ratio, 0.452.

From both Figure 18 and Figure 19, it can be seen that the tensile strength for Mix B is the highest at each age. It is important to note that Mix B was made with the same slump, 2.9 inches, as Mix A. In comparing Mix A and B, it is seen that the tensile strength for Mix B specimens is 119 percent at 3 days, 118 percent at 7 days, 116 percent at 14 days and 108 percent at 28 days, of the tensile strength of Mix A. This indicates that the admixture has the effect of increasing tensile strength of concrete if the slump is kept constant, in other words, the water/cement ratio is reduced by adding the admixture.

In this investigation, the amount of water for Mix B was 10 percent less than of Mix A but their cement and aggregate factors were unchanged. Then Mix B had the same slump, 2.9 inches, as Mix A.

Table IV.

Tension Test Results

Age in Days	Specimen Designation	Percentage Admixture Used	Maximum Load in pounds	Average Load in pounds	Average Tensile Strength in psi
3	A-1 A-2 A-3	0.00 0.00 0.00	30000 28900 29600	29500	261
3	B-1 B-2 B-3	0.25 0.25 0.25	39000 35500 30500	35000	310
3	C-1 C-2 C-3	0.25 0.25 0.25	32000 29000 32000	31000	274
3	D-1 D-2 D-3	0.50 0.50 0.50	31500 32000 35500	33000	292
7	A-4 A-5 A-6	0.00 0.00 0.00	41000 43300 42300	42200	374
7	B-4 B-5 B-6	0.25 0.25 0.25	51600 49000 48200	49600	440
7	C-4 C-5 C-6	0.25 0.25 0.25	42300 44100 43200	43000	380
7	D-4 D-5 D-6	0.50 0.50 0.50	45700 44000 46200	45300	398

Table IV.

(Continued)

Age in Days	Specimen Designation	Percentage Admixture Used	Maximum Load in pounds	Average Load in pounds	Average Tensile Strength in psi
14	A-7 A-8 A-9	0.00 0.00 0.00	48300 48700 52100	49700	440
14	B-7 B-8 B-9	0.25 0.25 0.25	59200 57800 59700	58900	521
14	C-7 C-8 C-9	0.25 0.25 0.25	50900 52300 51700	51600	456
14	D-7 D-8 D-9	0.50 0.50 0.50	54800 52800 53700	53800	476
28	A-10 A-11 A-12	0.00 0.00 0.00	59300 62000 56000	59100	523
28	B-10 B-11 B-12	0.25 0.25 0.25	65500 61500 63500	63500	562
28	C-10 C-11 C-12	0.25 0.25 0.25	52500 58000 57500	56000	496
28	D-10 D-11 D-12	0.50 0.50 0.50	61000 55500 56000	57500	510

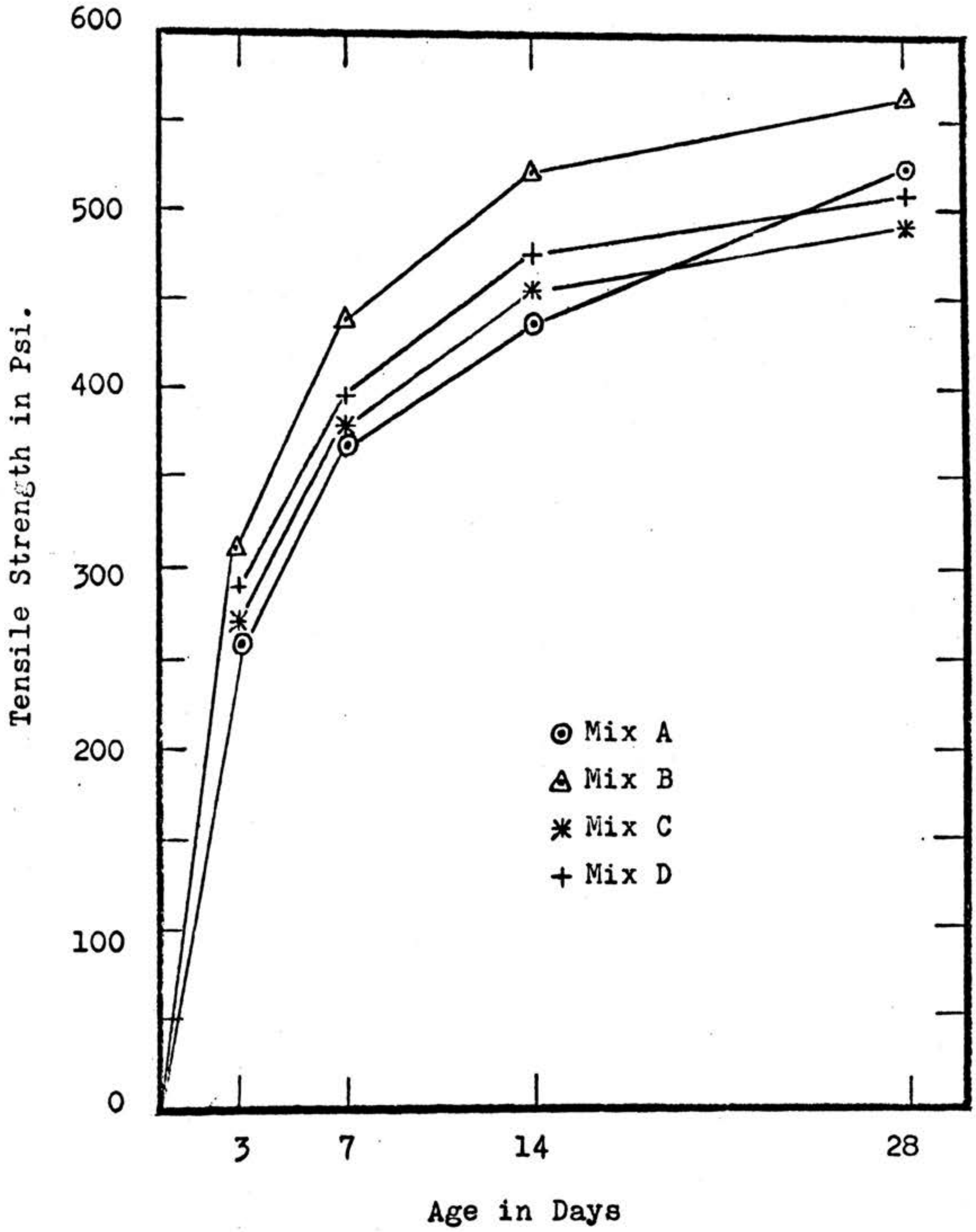


Figure 18. Tensile Strength Versus Age.

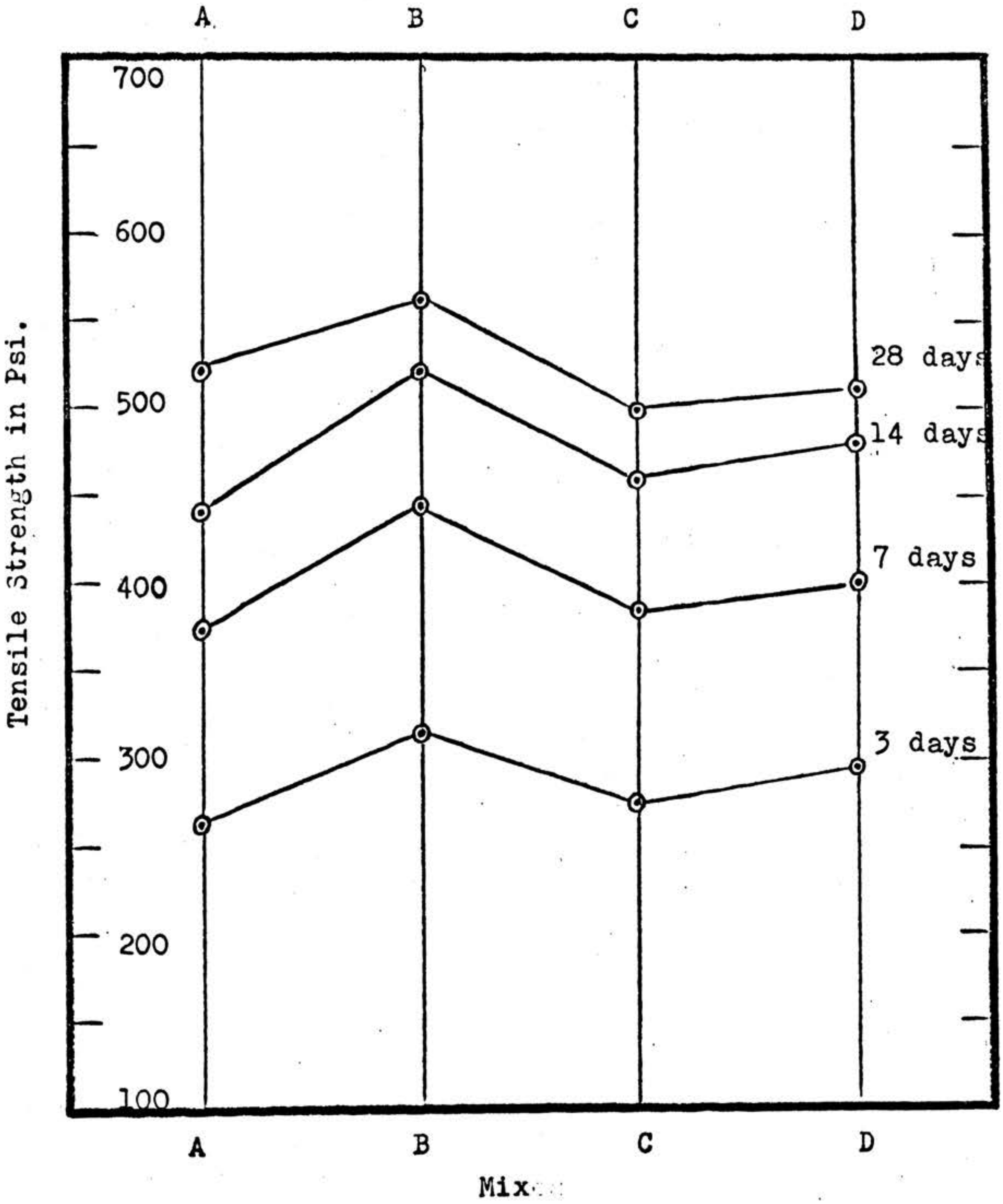


Figure 19. Tensile Strength Versus Mix

6.3 COMPRESSIVE STRENGTH TEST

The results of the compressive strength tests are shown in Table V. Figure 20 shows the relation between compressive strength and age for each mix, and Figure 21 shows the relation between compressive strength and mix for each age.

In comparing the 28 day strength of the four mixes A, B, C and D, from both Figure 20 and Figure 21, it is seen that Mixes B and D have higher strength than Mix A, however, the strength of Mix C is almost the same as Mix A.

From the figures it also can be seen that the compressive strength for Mix B is the highest at each age. Its strength is 125.5 percent at 3 days, 128 percent at 7 days, 126 percent at 14 days and 114 percent at 28 days, of the compressive strength of Mix A. This increase in compressive strength is believed to be due to the reduced water/cement ratio.

Table V.

Compression Test Results

Age in Days	Specimen Designation	Percentage Admixture Used	Maximum Load in pounds	Average Load in pounds	Average Compressive Strength in psi
3	A-1 A-2 A-3	0.00 0.00 0.00	75000 74000 80500	76500	2710
3	B-1 B-2 B-3	0.25 0.25 0.25	96000 95000 97000	96000	3400
3	C-1 C-2 C-3	0.25 0.25 0.25	77500 85500 77000	80000	2835
3	D-1 D-2 D-3	0.50 0.50 0.50	88000 92000 93000	91000	3220
7	A-4 A-5 A-6	0.00 0.00 0.00	93000 89000 92500	91500	3240
7	B-4 B-5 B-6	0.25 0.25 0.25	112000 104000 138000	118000	4180
7	C-4 C-5 C-6	0.25 0.25 0.25	96000 101000 94000	97000	3350
7	D-4 D-5 D-6	0.50 0.50 0.50	102000 105000 114000	107000	3780

Table V.

(continued)

Age in Days	Specimen Designation	Percentage Admixture Used	Maximum Load in pounds	Average Load in pounds	Average Compressive Strength in psi
14	A-7 A-8 A-9	0.00 0.00 0.00	121000 110000 99000	110000	3900
14	B-7 B-8 B-9	0.25 0.25 0.25	133000 147000 137000	139000	4930
14	C-7 C-8 C-9	0.25 0.25 0.25	110000 110000 125000	115000	4080
14	D-7 D-8 D-9	0.25 0.25 0.25	116000 131000 122000	123000	4360
28	A-10 A-11 A-12	0.00 0.00 0.00	134000 137000 134000	135000	4780
28	B-10 B-11 B-12	0.25 0.25 0.25	160000 149000 153000	154000	5440
28	C-10 C-11 C-12	0.25 0.25 0.25	137000 130000 135000	134000	4760
28	D-10 D-11 D-12	0.50 0.50 0.50	145000 140000 135000	140000	4960

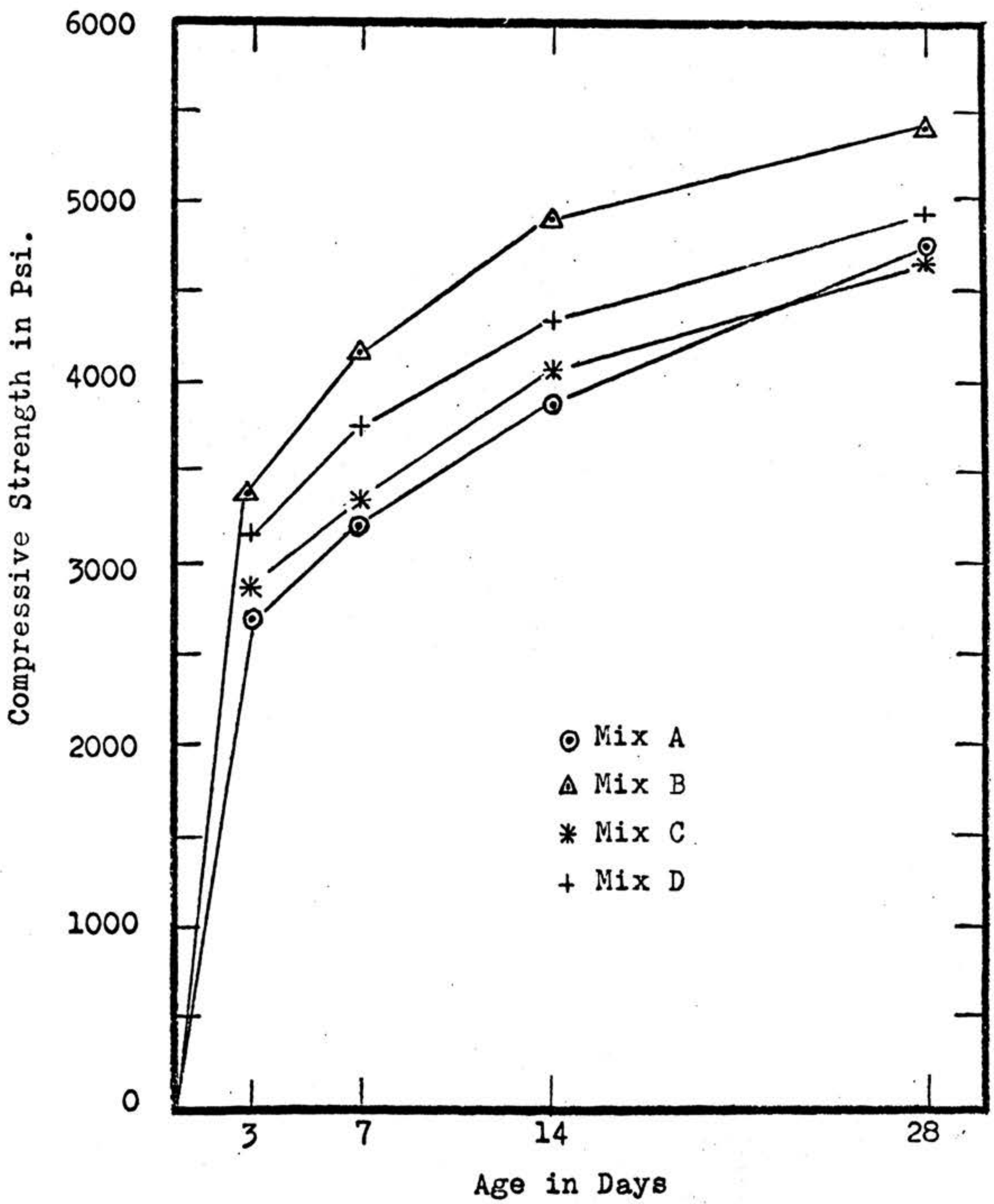


Figure 20. Compressive Strength Versus Age.

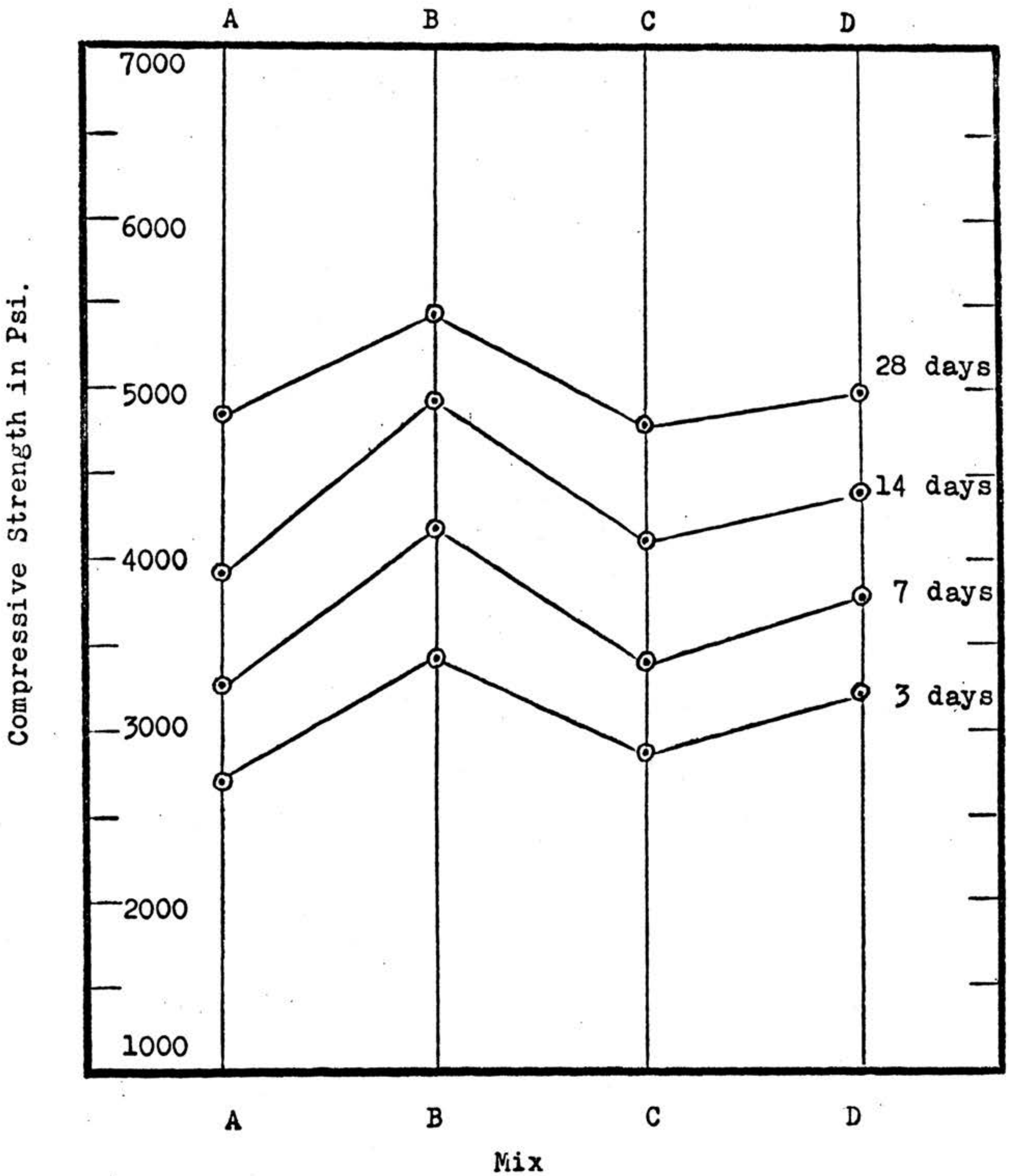


Figure 21. Compressive Strength Versus Mix

6.4 FLEXURAL STRENGTH TEST

The results of the tests on the flexural strength of concrete beams are shown in Table VI. Figure 22 shows the relation between modulus of rupture and age, and Figure 23 shows the relation between modulus of rupture and mix for each age.

Comparing the average 28 day strength of the three mixes A, C and D from the figures, it can be seen that the flexural strength of concrete decreased with increasing amounts of admixture if the water/cement ratio was kept constant, 0.452. This may also be due to the increasing air content in the mixes C and D.

The figures also indicate that the tendency to gain strength in all three cases is very nearly the same. Due to the reduced water/cement ratio, the flexural strength for Mix B became 114 percent at 3 days, 111 percent at 7 days, 104 percent at 14 days and 103 percent at 28 days of the flexural strength of Mix A.

Table VI.

Flexure Test Results

Age in Days	Specimen	Percentage Admixture Used	Maximum Load P in lbs	Modulus of Rupture R in psi	Average R in psi
3	A-1 A-2 A-3	0.00 0.00 0.00	2150 2100 2120	636 621 627	628
3	B-1 B-2 B-3	0.25 0.25 0.25	2400 2400 2450	710 710 725	715
3	C-1 C-2 C-3	0.25 0.25 0.25	2350 2400 2350	695 710 695	700
3	D-1 D-2 D-3	0.50 0.50 0.50	2500 2450 2550	740 725 755	740
7	A-4 A-5 A-6	0.00 0.00 0.00	2400 2450 2450	710 725 725	720
7	B-4 B-5 B-6	0.25 0.25 0.25	2750 2700 2650	815 800 785	800
7	C-4 C-5 C-6	0.25 0.25 0.25	2550 2500 2570	755 741 760	752
7	D-4 D-5 D-6	0.50 0.50 0.50	2580 2650 2580	764 785 764	771

Table VI.

(Continued)

Age in Days	Specimen	Percentage Admixture Used	Maximum Load P in lbs	Modulus of Rupture R in psi	Average R in psi
14	A-7 A-8 A-9	0.00 0.00 0.00	2800 2870 2850	829 850 844	841
14	B-7 B-8 B-9	0.25 0.25 0.25	2920 3000 2960	865 888 875	876
14	C-7 C-8 C-9	0.25 0.25 0.25	2770 2720 2800	820 805 829	818
14	D-7 D-8 D-9	0.50 0.50 0.50	2950 2750 2700	873 815 799	829
28	A-10 A-11 A-12	0.00 0.00 0.00	3200 3300 3100	946 976 916	946
28	B-10 B-11 B-12	0.25 0.25 0.25	3350 3200 3340	990 946 989	975
28	C-10 C-11 C-12	0.25 0.25 0.25	3150 3000 3150	932 888 931	917
28	D-10 D-11 D-12	0.50 0.50 0.50	3100 3000 3040	916 888 900	901

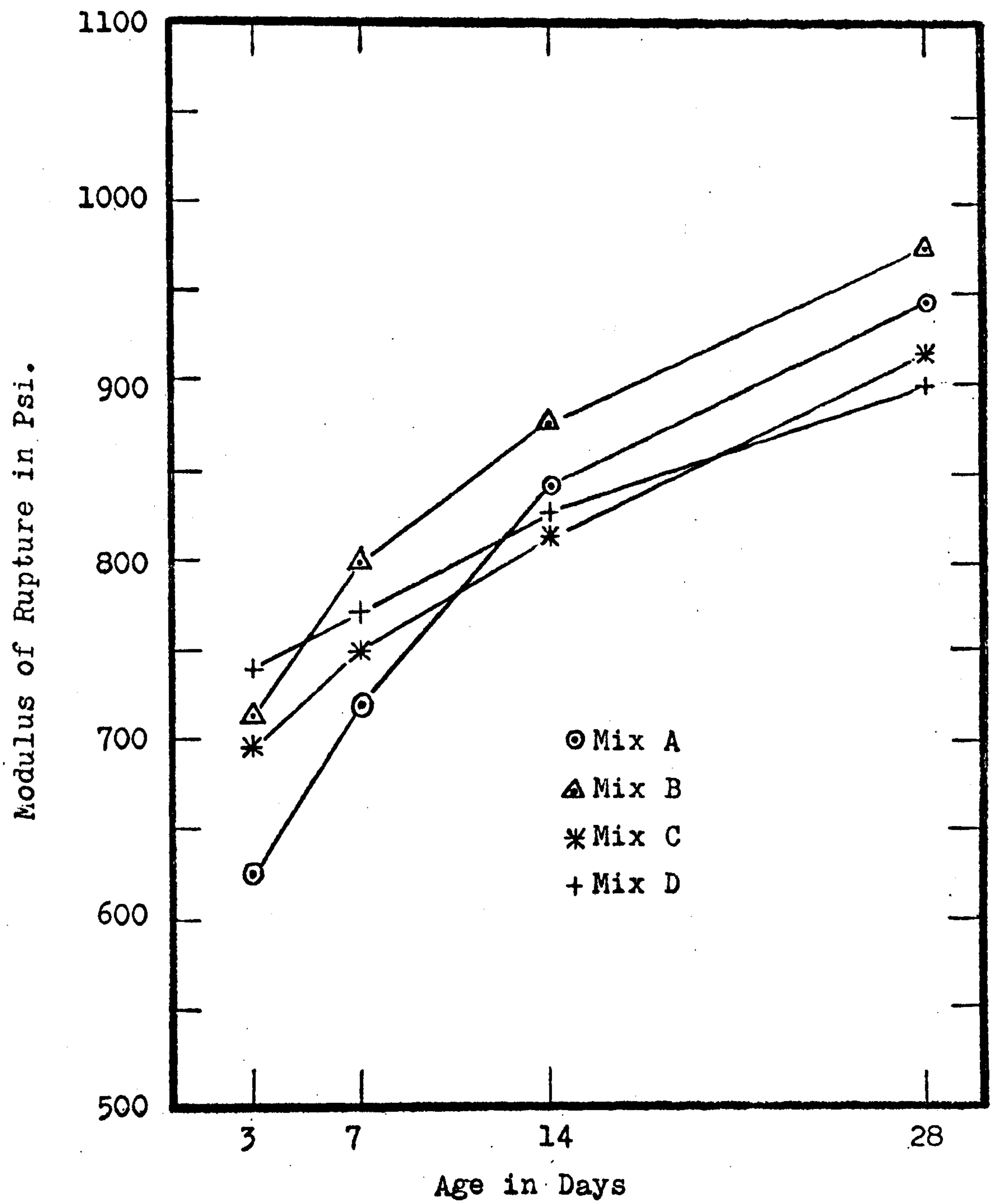


Figure 22. Modulus of Rupture Versus Age.

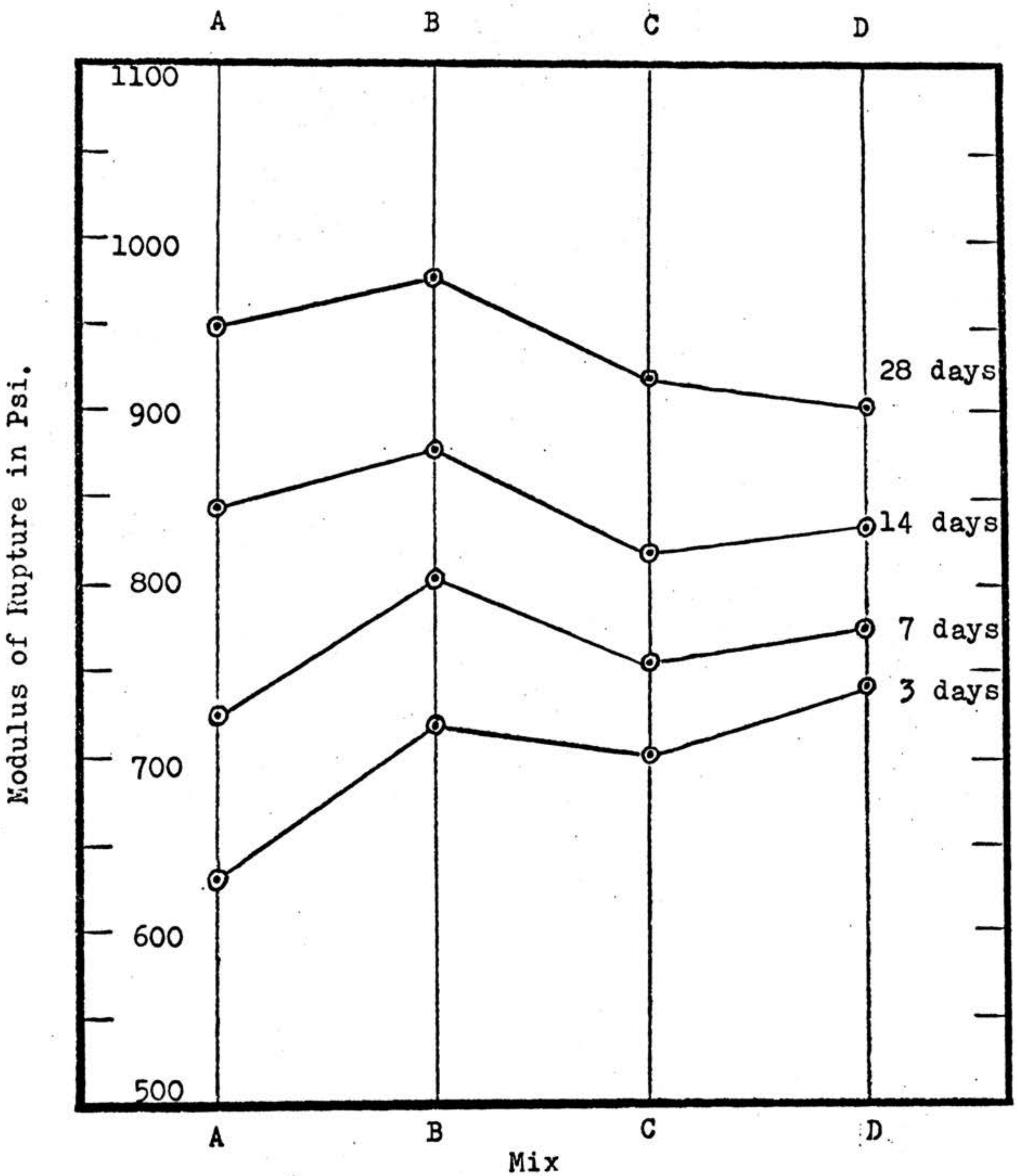


Figure 23. Modulus of Rupture Versus Mix

6.5 SHRINKAGE TEST

In this test, specimens were kept for 28 days in a moist room and then 7 days in laboratory air. All specimens were measured using a micrometer capable of reading to the nearest one-thousandth of an inch.

The results of the shrinkage test are shown in Table VII. From the table it can be seen that after 35 days the average shrinkage was a constant for the A, C and D specimens. There was a little swelling of the B specimens. However, there was no observed effect in this shrinkage test. It is necessary to determine the effect of the admixture on shrinkage for a longer curing period.

Table VII.

Shrinkage Test Results

Specimen Number	Length at 1 Day inches	Length at 28 Days inches	Length at 35 Days inches	Shrinkage (-) or Swelling (+) percent
A-1	11.800	11.800	11.800	------
A-2	11.619	11.618	11.618	-0.009
A-3	11.526	11.525	11.525	-0.009
B-1	11.522	11.523	11.523	+0.009
B-2	11.667	11.667	11.667	------
B-3	11.813	11.813	11.813	------
C-1	11.795	11.794	11.794	-0.009
C-2	11.450	11.450	11.450	------
C-3	11.516	11.516	11.516	------
D-1	12.181	12.180	12.180	-0.009
D-2	11.521	11.520	11.520	-0.009
D-3	12.486	12.486	12.486	------

VII. SUMMARY AND CONCLUSIONS

7.1 SUMMARY

In this investigation, calcium lignosulfonate was used as an admixture. Four batches of concrete with varying amounts of admixture and water/cement ratios were tested for tensile strength, compressive strength, flexural strength, shrinkage, air content and slump. The water/cement ratio selected and the percentages of admixture used for Mixes A, B, C and D are shown in Table III, the Mix A being the standard of comparison.

It was found if the water/cement ratio is kept constant while adding calcium lignosulfonates, both air content and slump increased and also the tensile and flexural strengths of concrete decreased.

It was also found that if the workability, as measured by the slump test, is kept constant, the water/cement ratio is reduced by adding calcium lignosulfonates. The reduction in the water/cement ratio results in an increase in the strengths of concrete.

7.2 CONCLUSIONS

The results of this series of tests show that the effect of calcium lignosulfonate upon the physical properties of a concrete mixture is very pronounced. The following conclusions have been made either from the test results or by observation.

1. Calcium lignosulfonate reduces the amount of water required to make a workable concrete mix-- in some instances

up to 10 percent.

2. A more workable mix saves money because it is easier to place, reducing labor requirements for the project. Using calcium lignosulfonate, it is possible to increase slump and air content and at the same time increase the strength by reducing the water/cement ratio.

3. At the same slump, the concrete containing calcium lignosulfonate will be more plastic and may be placed and consolidated more easily.

4. Reducing the water/cement ratio while leaving the cement factor unchanged results in concrete of higher tensile, compressive and flexural strengths at all ages. At a given cement factor, tensile strength gains of 17.7 percent at 7 days and 7.5 percent at 28 days, compressive strength gains of 28.1 percent at 7 days and 13.6 percent at 28 days, flexural strength gains of 11.1 percent at 7 days and 3.1 percent at 28 days, were obtained.

5. Most high performace concrete is designed with about 5 percent entrained air to increase freeze-thaw durability. While calcium lignosulfonate is not considered to be an air-entraining agent, it entrains just a little air over plain concrete at the same slump. However, only half the normal dosage of air-entraining agent may give the desired air content when calcium lignosulfonate is used.

6. Excess mixing water is the major cause of drying shrinkage in concrete, though cement content, air content, and the nature of the aggregate also have an effect. Since calcium

lignosulfonate reduces the total water requirements, the drying shrinkage tendency should be less at all ages althougth there was no observed effect on this shrinkage test.

7. At the same water/cement ratio, the concrete containing calcium lignosulfonate will decrease the tensile strength and flexural strength.

7.3 RECOMMENDATIONS

The results of this investigation indicate that high early tensile, compressive and flexural strengths can be obtained by using calcium lignosulfonate as an admixture, by reducing the water content while leaving the cement factor unchanged. The author recommends that a study of the effect of this admixture in prestressed concrete be conducted. In prestressed concrete, normally a prestressing force is applied at early ages. It is very important for prestressed concrete to have high early strength.

Because of the increase in slump when using calcium lignosulfonate, it is possible to reduce the cement factor by some quantity and still obtain strengths higher than the control mix with an unreduced cement factor, giving the same quality of concrete at lower cost. Therefore, the author would like to recommend that a study of the effect of reducing the cement factor on strengths be conducted.

Since it is possible to reduce the cement factor, it is also possible to reduce heat of hydration of concrete. In addition the author recommends that it is necessary to test the effect of calcium lignosulfonate on heat of hydration.

There may be good agreement that the use of this admixture reduces the heat of hydration in concrete which is an important consideration in the construction of large dams.

The test results show that additional insight into the use of admixtures could be gained if these tests were varied and extended. A more detailed investigation consisting of varying the percentage of admixture used, at the same slump, may delineate the point of transfer from increasing strength to decreasing strength in concrete, if any.

Further tests should be run to determine the effect of the admixture on properties of concrete for a longer curing period.

BIBLIOGRAPHY

1. Bruce Foster, "Effect of Water-Reducing Admixtures and Set-Retarding Admixtures on Properties of Concrete," Symposium On Admixtures In Concrete, ASTM Special Technical Publication, 1959, pp. 1-2.

2. Ugur Ozoguz, "Properties of Air-Entrained Concrete at Early Ages," Thesis, University of Missouri School of Mines and Metallurgy, Rolla, Missouri, 1960, pp. 1-3.

3. Orchard, "Concrete Technology" Vol. 1, 1962, pp. 81-129.

4. Lerch, W. "The Effect of Added Materials on the Rate of Hydration of Portland Cement Pastes at Early Ages," Portland Cement Association, Research Report, August 1943 to January 1944.

5. Grieb, W. E., Werner, G. and Woolf, D. G. "Water-Reducing Retarders for Concrete-Physical Tests," Public Roads, Vol. 31, No. 6, February 1961.

6. "Pigments for Colouring Cement and Concrete," British Standard 1014, 1942, British Standards Institution.

7. Klieger P. "Studies of the effect of Entrained Air on the Strength and Durability of Concretes made with Various Maximum Sizes of Aggregate," Proceedings of the Highway Research Board, Vol. 31, 1952.

8. Lewis H. Tuthill, Robert F. Adams, and John M. Hemme, Jr. "Observations in Testing and Use of Water-Reducing Retarders." ASTM Special Technical Publication No. 266, 1956.

9. "Marathon, Chemicals from Wood," Bulletin No. 130. Pamphlet of American Can Company, Neenah, Wisconsin.

10. "Design and Control of Concrete Mixtures, Tenth Edition." Portland Cement Association's Pamphlet, pp. 4-19.

11. Wright, P.J.F., "Comments on an Indirect Tensile Test on Concrete Cylinders," Magazine of Concrete Research, Vol. 7, No. 20, July 1955, pp. 87-96, London, England.

APPENDICES

APPENDIX A

Appendix A contains the concrete mix designs utilized to obtain the experimental cylinders and beams. The method of design is based upon the Portland Cement Association water/cement ratio design criteria.

1. MIX DESIGN DATA

Maximum Size of Gravel: 3/4 inch
Slump of Concrete Selected: 3 inches
Specific Gravity of Gravel: 2.65
Specific Gravity of Sand: 2.55
Specific Gravity of Portland Cement: 3.15
Fineness Modulus of Sand: 2.60--Medium
Unit Weight of Water: 62.4 lb/cu. ft.
Total Water Used per Cu. Yd.: 38 gal
Water per Sack of Cement: 5.5 gal
One Sack of Cement Weighed: 94 lbs
One Gallon of Water Weighed: 8.33 lbs
Free Moisture of Sand: 1.25 percent
Free Moisture of Gravel: 0.50 percent
Fine Aggregate --% of Total Aggregate: 46

2. MIX DESIGN PROCEDURE

Gallons Water/Sack of Cement

Cement Factor: $\frac{\text{Net Gallons Water}}{\text{Gal./Sack Cement}}$

Absolute Volume of Cement: $\frac{\text{(Wt./Bag of Cement)(Cement Fact)}}{\text{(S. G. Cement)(Unit Weight } H_2O\text{)}}$

Absolute Volume Water: $\frac{\text{Net Gallons Water}}{\text{Gal./Cu. Ft. Water}}$

Volume Paste: Volume Cement + Volume Water

Absolute Volume Aggregate: Cu. Ft./Yard - Volume Paste

Absolute Volume Sand: (Volume Aggregate)(% Sand)

Weight Dry Sand: (Volume Sand)(Unit Wt. Water)(S. G. Sand)

Absolute Volume Gravel: (Volume Aggregate)(% Gravel)

Weight Dry Gravel: (Vol. Gravel)(Unit Wt. Water)(S.G. Gravel)

Moisture Correction

Moisture Content of Sand

Moisture Content of Gravel

Moisture in Sand: (% Moisture)(Weight Sand)

Moisture in Gravel: (% Moisture)(Weight Gravel)

Total Free Water: (Moisture in Sand) + (Moisture in Gravel)

Cubic Yard Mix Proportions

Wt. of Moist Sand: (Wt. of Surface Dry Sand)(1 + M.C.)

Wt. of Moist Gravel: (Wt. of Surface Dry Gravel)(1 + M.C.)

Water: (Net Gallons Water)(Wt. of Water per Gallons)
- Moisture Correction

Cement: (Wt. of Cement per Bag)(Cement Factor)

Mix Ratio

Cement : Sand : Gravel

3. MIX DESIGN

Gal. H_2O/Sack Cement: 5.5

Cement Factor: 38/5.5 = 6.91

Abs. Vol. Cement: $\frac{(94)(6.91)}{(3.15)(62.4)}$ = 3.3 cu. ft.

Abs. Vol. Water: 38/7.48 = 5.07 cu. ft.

Vol. Paste: 3.3 + 5.07 = 8.37 cu. ft.

Abs. Vol. Agg.: 27 - 8.37 = 18.63 cu. ft.

Abs. Vol. Sand: (0.46)(18.63) = 8.57 cu. ft.

Wt. Dry Sand: (62.4)(8.57)(2.55) = 1365 lbs

Abs. Vol. Gravel: (1 - 0.46)(18.63) = 10.07 cu. ft.

Wt. Dry Gravel: (62.4)(10.07)(2.65) = 1665 lbs

Moisture Correction

Moisture Content of Sand: 1.25%

Moisture Content of Gravel: 0.50%

Moisture in Sand: (0.0125)(1365) = 17.1 lbs

Moisture in Gravel: (0.005)(1665) = 8.33 lbs

Total Free Water: 17.1 + 8.33 = 25.43 lbs

Cubic Yard Mix Proportions

Wt. of Moist Sand: (1365)(1 + 0.0125) = 1380 lbs

Wt. of Moist Gravel: (1665)(1 + 0.005) = 1673.3 lbs

Water: (38)(8.33) - (25.43) = 290.77 lbs

Cement: (94)(6.91) = 649 lbs

Mix Ratio

Cement : Sand : Gravel = 1 : 2.13 : 2.58

APPENDIX B

The quantities of cement, aggregate and water used for the different batches are given below.

BATCH A

	POUNDS
Cement	69.50
Coarse aggregate	148.50
Fine aggregate	179.70
Water	31.40
Calcium lignosulfonate	0.00

BATCH B

	POUNDS
Cement	69.50
Coarse aggregate	148.50
Fine aggregate	179.70
Water	28.26
Calcium lignosulfonate	0.17

BATCH C

	POUNDS
Cement	69.50
Coarse aggregate	148.50
Fine aggregate	179.70
Water	31.40
Calcium lignosulfonate	0.17

BATCH D

	POUNDS
Cement	69.50
Coarse aggregate	148.50
Fine aggregate	179.70
Water	31.40
Calcium lignosulfonate	0.35

VITA

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He received his primary education at the Chung-Cheng Primary School, and received his secondary education at the Middle School of Taiwan Provincial Normal University, Taipei, Taiwan, China.

In June, 1962, he received a Bachelor of Science degree in Civil Engineering from Taiwan Provincial Cheng Kung University, Tainan, Taiwan, China.

From August, 1962, to August, 1963, he served in the Chinese Army as a Second Lieutenant.

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In the Fall of 1964 he came to the United States of America, and transferred to the University of Missouri at Rolla, Missouri, to continue his studies for the degree of Master of Science in Civil Engineering.